ARL-TN-0858 ● Nov 2017

US Army Research Laboratory

Influence of Thermal Annealing and a Glass Coating on the Strength of Soda-Lime-Silicate Glass

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REPORT DOCUMENTATION PAGE	*Form Approved* *OMB No. 0704-0188*

1. REPORT DATE *(DD-MM-YYYY)*	**2. REPORT TYPE**	**3. DATES COVERED (From - To)**
November 2017	Technical Note	31 August 2012–01 September 2013

4. TITLE AND SUBTITLE
Influence of Thermal Annealing and a Glass Coating on the Strength of Soda-Lime-Silicate Glass

5a. CONTRACT NUMBER

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)
Mariel H Gaviola, Steven Kilczewski, Jeffrey J Swab, Parimal J Patel, and Luke Gilde

5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
US Army Research Laboratory
Weapons and Materials Research Directorate (ATTN: RDRL-WMM-E)
Aberdeen Proving Ground, MD 21005-5066

8. PERFORMING ORGANIZATION REPORT NUMBER
ARL-TN-0858

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

10. SPONSOR/MONITOR'S ACRONYM(S)

11. SPONSOR/MONITOR'S REPORT NUMBER(S)

12. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution is unlimited.

13. SUPPLEMENTARY NOTES

14. ABSTRACT
This project investigated the influence that thermal annealing and the addition of a glass coating has on the flexure strength of a soda-lime-silicate glass used in a variety of military ballistic armor applications. A mixture of glass powder and isopropyl alcohol was sprayed onto the tin side of as-manufactured soda-lime-silicate glass plates and on plates with a 10-mm-long scratch created with either a 1- or 10-N scratch load. The glass coating was cured using an annealing cycle of 6 °C/min from room temperature to 675 °C, followed by a 1.5-h hold at temperature, and then cool down to room temperature at 3 °C/min. Plates that were scratched showed a noticeable strength increase following the annealing process; however, the addition of the glass coating did not result in an additional strength increase for the scratched plates.

15. SUBJECT TERMS
soda-lime-silicate glass, flexure strength, glass coating, thermal annealing, strength recovery

16. SECURITY CLASSIFICATION OF:			**17. LIMITATION OF ABSTRACT**	**18. NUMBER OF PAGES**	**19a. NAME OF RESPONSIBLE PERSON**
					Jeffrey J Swab
a. REPORT	**b. ABSTRACT**	**c. THIS PAGE**			**19b. TELEPHONE NUMBER (Include area code)**
Unclassified	Unclassified	Unclassified	UU	16	(410) 306-0753

Standard Form 298 (Rev. 8/98)
Prescribed by ANSI Std. Z39.18

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Acknowledgements

I would like to thank Dr Jeffrey Swab for the privilege of researching ceramics materials under his mentorship throughout my Capstone Project. I am incredibly grateful for his patience and enthusiasm in furthering my engineering career. I would like to thank Mr Jared Wright and Mr Steve Kilczewski for their partnership and guidance. I would like to thank Elizabeth Wilson, Ashley Gott, and Blake Paulson for their support in my research endeavors. I would like to thank the US Army Research Laboratory, Dr Sandra Young, and the Science and Engineering Apprenticeship Program for providing me with this valuable experiential learning.

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1. Introduction

Glass is a common constituent in many transparent armor systems on military vehicles. Based on the strength of the highly covalent silicon–oxygen bonds that comprise a typical glass, it has been calculated that the intrinsic strength of a flawless glass is around 35 GPa.[1] Unfortunately, this high strength is rarely, if ever, achieved since imperfections introduced to the glass surface because processing and handling drastically limit the strength potential. Furthermore, the environment, specifically the water vapor in the atmosphere, interacts with these imperfections to contribute to the lower strength.[2] Various methods have been utilized to improve strength by introducing a compressive stress on the glass surface. This includes traditional methods such as ion exchange[3,4] and thermal annealing and tempering.[5,6] The compressive stress generated by these processes increases resistance to crack initiation and propagation and consequently the overall strength. Acid etching[7] and the more recent use of laser[8] and plasma-arc[9] surface treatments have been shown to minimize the severity of these surface imperfections, resulting in higher strength values. Most of these methods are not practical or economical for strengthening the large pieces of glass used in many applications, including transparent armor. An alternative option is to coat the glass surface to prevent imperfections from forming, especially those due to handling. As a result, existing flaws can be minimized and environmental effects can be mitigated. Several studies have used sol-gel-based coatings with varying degrees of success.[10–12] This report summarizes an effort to determine the extent of strengthening imparted on a soda-lime-silicate glass using a thermal anneal as well as a thermal anneal coupled with a glass coating.

2. Experiment Procedure

Plates of a low-iron soda-lime-silicate glass (trade name Starphire) from PPG (PPG Industries, Inc., Pittsburgh, Pennsylvania) were obtained with a nominal size of 100 × 100 × 6 mm. The plates were separated into 3 sets: baseline, annealed, and annealed plus glass coated. Within each set, 3 subsets were created: as-received, 1-N scratched, and 10-N scratched. The latter 2 subsets had a single scratch introduced in the relative center of the tin side of each plate. A 10-mm-long guideline was drawn on the center of the tin side of each glass plate with permanent marker. The appropriate mass for a 1- or 10-N load was then placed on the beam of the Elcometer 3086 Scratch Boy (Fig. 1). The tip of the diamond scribe attached to the unit was placed in contact with the glass surface at one end of the guideline and then mechanically pulled across the plate following the guideline to generate the scratch. Once the end of the guideline was reached, the tip of the scribe was lifted off the glass.

Fig. 1 Elcometer 3086 Scratch Boy with diamond scribe

Annealing was completed by placing the glass plates, tin side up, in a firing kiln on thin, individual, high-density alumina setter tiles of the same size to minimize any reaction between the glass and the furnace lining (Fig. 2). A low-density setter plate was elevated on the bricks on the right- and left-hand sides of the kiln above the plates to cover the arrangement and minimize the likelihood of particles from the kiln's refractory lining landing on the glass plates during the annealing cycle. The firing kiln held 9 glass plates per annealing cycle. The prescribed cycle for all annealed plates was the following: ramp up from room temperature to 675 °C at a rate of 6 °C/min, followed by a 1.5-h hold at 675 °C, and then ending with a controlled cool down to room temperature at 3 °C/min.

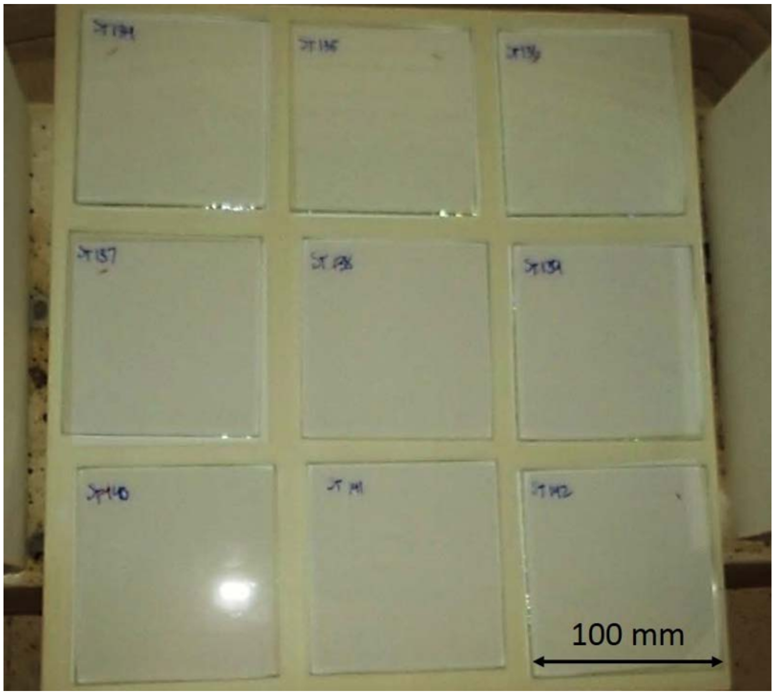

Fig. 2 Firing kiln setup used for annealing and creation of the glass coating. Each glass plate is resting on top of a thin alumina setter plate 100 mm square.

A glass coating was created on the tin surface of baseline and scratched glass plates by spraying a glass slurry comprised of SP-188 glass powder (distributed by Specialty Glass, Inc.) and isopropyl alcohol (IPA). Annealing was conducted at the temperatures and times listed previously. The slurry was prepared to provide a solids loading of 50% based on the density of the SP-188 and the IPA. Application of the slurry was completed in a fume hood to minimize release of the spray into the laboratory atmosphere. Ultra-high-purity nitrogen at a static pressure of 50 psi, yielding a dynamic pressure of 35 psi, combined with a SATA spray gun, transferred the glass slurry onto the tin surface of the glass (Fig. 3). Plates were individually sprayed until 3.5 ± 0.10 g of slurry was deposited on the tin surface. To reduce overspray and potential adherence of the glass plate to the alumina setter tile during the firing cycle, the uncoated air side and all edges of each glass plate were wiped with damp KimWipes to remove excess slurry before placement into the firing kiln. Nine plates were placed in the kiln, and the same thermal cycle used for the anneal experiments was used to heat the plates and convert the slurry into a glass coating.

Fig. 3 Glass slurry being sprayed onto the tin surface of a glass plate

In preparation for equibiaxial flexure strength testing, the thickness at the 4 corners of each glass plate was measured with a micrometer, and the average thickness was determined and used in the calculation of the plate strength. A transparent low-tack adhesive layer, measuring approximately 100 × 100 × 0.005 mm, was placed on the side in compression (air side) prior to strength testing to help prevent pieces of glass from be ejected when the plate fractured. Grafoil squares measuring 100 × 100 × 0.127 mm were placed on the air and tin sides to provide a near-frictionless surface and improve stress distribution during testing. Once the plate fractured, the maximum break force (measured in Newtons) was recorded and used to determine the plate strength. A minimum of 15 plates were tested at each condition to calculate the average equibiaxial flexure strength and to determine the impact of annealing and glass coating on the strength. All equibiaxial flexure strength testing was conducted following the procedures outlined in ASTM C1499,[13] using load and support rings with 42.5- and 85-mm diameters (a ratio of 0.5), respectively, and a loading rate of 7.6 mm/min.

3. Results and Discussion

The results in Table 1 summarize the strength of the tin side of the Starphire soda-lime-silicate glass as a function of scratch load, thermal annealing, and thermal annealing with a glass coating.

Table 1 Strength summary[a]

Plate	Baseline			Annealed			Annealed and glass coated		
	n	σ (MPa)	StD	n	σ (MPa)	StD	n	σ (MPa)	StD
As-received	27	132.7	24.0	18	162.5	42.5	16	79.9	14.5
1-N scratch	20	62.1	20.8	31	103.8	19.3	12	98.4	17.5
10-N scratch	44	35.1	7.3	35	83.9	20.1	13	80.5	21.4

[a]Tin side in tension for all tests.
Note: StD = standard deviation.

The strength decreased when a scratch was introduced on the tin side of each Starphire plate. A decrease in strength of approximately 50% is observed when a 1-N scratch is introduced on the glass surface, and then an additional 40% decrease is seen when a 10-N scratch is introduced. This is slightly different from the strength decrease observed in a different soda-lime-silicate glass[14] when scratches of the same size and magnitude were placed on the tin side. The difference in the latter case was that the strength dropped to approximately 35 MPa when the 1-N scratch load was used, and the strength remained consistently around 35 MPa even as the scratch load increased to 30 N.

Thermal annealing resulted in a strength increase for the baseline Starphire as well as 1- and 10-N scratched plates. The strength increase measured in the scratched plates was much more dramatic than the strength increase of the baseline glass. The strength increase in the baseline glass was minimal since the increase was associated with a significant increase in standard deviation. The thermal annealing process is designed to blunt or heal flaws. It appears that the annealing process used in this study was sufficient to heal/blunt many of flaws associated with the 10-mm-long scratches but it seems to be insufficient to heal/blunt all of the microscopic (not seen with the naked eye) flaws distributed across the entire surface of each plate.

Thermal annealing coupled with the addition of a glass coating did not result in any further strengthening of the scratched plates. Surprisingly, it appears to have reduced the strength of the baseline glass. This apparent strength reduction, along with the lack of additional strengthening of the scratched plates, may be due to a lack of uniformity and to the presence of bubbles in the applied glass coating. Since the glass/IPA mixture was deposited on each plate by hand, it is possible that the resulting glass-coating thickness was not uniform. Such inconsistencies could lead to nonuniform stress distribution across the plate surface during strength testing. The glass coating was typically translucent, not transparent, on the plate after curing indicating the presence of bubbles in the coating. On some plates the density of bubbles was clearly not uniform, as there were observable dark gray regions (Fig. 4). These dark regions are typically due to a large concentration of bubbles that do not permit light to transmit through the coating (bubble opacity). A 10–15 °C increase in the annealing temperature may be sufficient to reduce the viscosity of the glass coating enough to enable the development of a uniformly thick coating and permit the bubbles to fine out. Further research is needed to confirm this hypothesis.

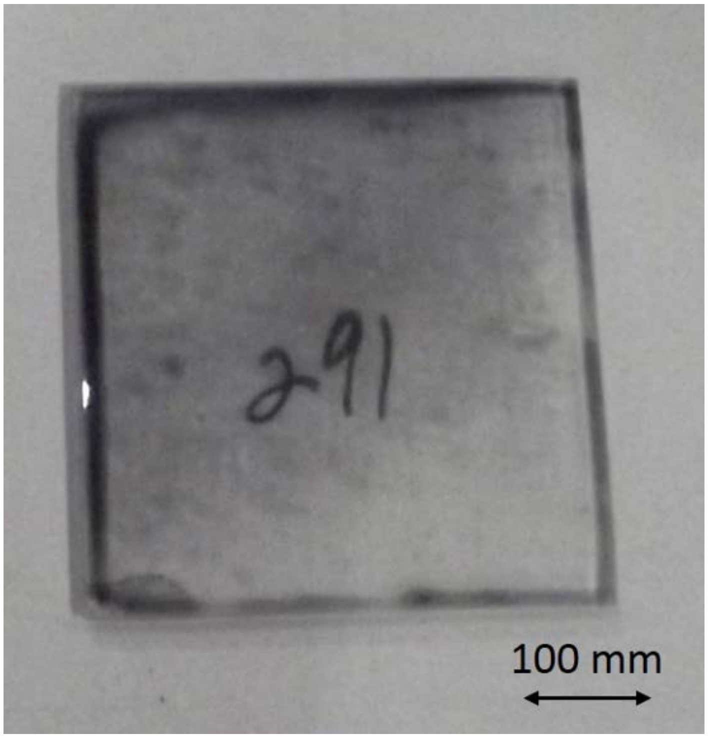

Fig. 4 Example of the gray color variation seen in the glass coating

4. Summary and Conclusion

The equibiaxial flexure strength of a low-iron-containing soda-lime-silicate glass (Starphire) was determined after scratches were introduced on the tin surface of plates. Attempts were made to minimize the impact of these scratches by thermal annealing as well as by thermal annealing coupled with a glass coating. Introducing a scratch reduced the strength significantly, but a large portion of this strength could be recovered when the plates were exposed to a thermal anneal. The addition of a glass coating in tandem with the thermal anneal process did not provide any additional strengthening due to the presence of bubbles in the glass coating. A slight increase in the annealing temperature may be sufficient to reduce the coating viscosity and enable the bubbles to fine out, potentially contributing to additional strengthening.

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